Saskatchewan
Agriculture, Food
and Rural
Revitalization

ADF

AGRICULTURE DEVELOPMENT FUND

FINAL REPORT

19990394

SEA BUCKTHORN OIL EXTRACTION USING COLD PRESS CENTRIFUGE

Funded by: Agriculture Development Fund

November 2000

Prepared by: Creekside Acres

106-01223/

FINAL REPORT

TO

AGRICULTURE DEVELOPEMENT FUND

EXTRACTION OF OIL FROM SEA BUCKTHORN BERRIES

Submitted by

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SUMMARY

Creekside Acres approached P.O.S. Pilot Plant to carry out solvent and non-solvent extraction trials in order to isolate the oil from Sea Buckthorn berries. The objectives were:

- To evaluate a non-solvent oil isolation protocol (lab scale)
- To carry out solvent oil extraction trials (pilot scale)
- To produce crude oil

The crude oil produced from the pilot scale was analyzed for specific gravity, pH, viscosity, fatty acid composition, tocopherols, vitamin E, carotene and residual hexane content. The solvent extraction method obtained higher oil yields than the non-solvent method, however the non-solvent process was more extensive and the oil quality was less.

TECHNICAL REPORT

Please refer to the attached Final Report to Creekside Acres from P.O.S. Pilot Plant Corp.

The impact of this research is crucial to the identification of an efficient process for oil extraction to support the international demand for Sea Buckthorn oil products.

ACKNOWLEDGEMENT

The Saskatchewan Agriculture and Food Department largely provided funding for this project.

Creekside Acres would not have been able to undertake this project if not for the financial support by Saskatchewan Agriculture and Food. Diversification is essential to the survival of agriculture in this province, we are pleased with opportunity to contribute this progress to the industry and future markets.

August 4 2000

FINAL REPORT

TO

CREEKSIDE ACRES

Extraction of oil from Sea Buckthorn Berries

Submitted by

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SUMMARY

Creekside Acres approached POS to carry out solvent and non-solvent extraction trials in order to isolate the oil from the sea buckthorn berry. A non solvent oil isolation protocol defined by Creekside Acres was evaluated at the lab scale. Solvent (hexane) extraction trials were carried out at the pilot scale. Approximately 99 kg of whole sea buckthorn berries were processed. The berry contains, on a wet basis, 2.37% oil. Oil yield obtained from the solvent and the non-solvent extraction trials was 2.08%, and 0.19% (oil/starting berries) respectively. The crude oil produced from the pilot scale trials was analyzed for specific gravity, pH, viscosity, fatty acid composition, tocopherols, vitamin E, carotene, and residual hexane content.

INTRODUCTION

Creekside Acres requested POS to carry out lab and pilot scale trials to extract oil from sea buckthorn berries

Sea buckthorn is a unique plant currently being domesticated. The three main sources of valuable products are derived from the berries, juice, pulp and oil. The juice is high in vitamins and carotene, and thus provides a nutritious beverage. The pulp provides for extraction of "sea buckthorn yellow" pigment that has potential use as food colouring material. Finally, the oil shows promise, because of its light absorption and emollient properties, as an ingredient in cosmetics, phytopharmaceutiacls , or UV skin protectant preparations (Beveridge *et al.*, 1999).

Oil can be separated from the sea buckthorn berry through solvent or non-solvent extraction. Solvent method uses one or several organic solvents to extract the oil from whole or berry components. Non-solvent method usually applies centrifugal force to separate the oil from the berry components based on their specific gravity differences.

Isolation of sea buckthorn oil using solvent and non-solvent methods were both evaluated in the current project. Pilot scale processing equipment was identified.

OBJECTIVES

The objectives of this project were:

- To evaluate, at the lab scale, a non-solvent oil isolation protocol.
- To carry out, at the pilot scale, solvent (hexane)oil extraction trials.
- To produce crude sea buckthorn oils for Creekside Acres.

MATERIALS AND METHODS

Approximately 99kg of frozen sea buckthorn berries were provided be Creekside Acers. The berries were grown in Saskatchewan and harvested in the winter 1999-2000. Approximately 3 kg of the berries were used for lab studies, and the remaining 96kg of berries were used for the pilot scale oil extraction trials.

1. Lab scale non-solvent oil isolation trials

Prior to non-solvent isolation trials, lab work was carried out in order to determine basic information on the sea buckthorn berry, such as moisture and oil content , and its weight composition.

1.1. Determination of moisture

Approximately 30kg of whole berries were oven dried to dryness at 130 degrees Celsius for approximately 3 hr. Weight differences before and after the drying were recorded, and moisture content of the whole berry was calculated.

1.2. Determination of weight composition

Approximately 473g of whole berries were macerated, and screened to separate crude juice from seeds and pulp. The seeds were manually separated from the pulp, and oven dried at 130 degrees Celsius for 1,5hrs. The pulp was combined with the crude juice, and oven dried to dryness at 130 degrees Celsius for approximately 3hrs. The dried seeds and pulp were weighed, and their weight composition as berry components was determined.

1.2. Determination of oil contents

Approximately 30g of whole berries were oven dried to dryness at 130 degrees Celsius for approximately 3 hrs. The dried whole fruits were ground and solvent (hexane) extracted simultaneously on a mechanical extractor system. The oil extracted was weighed and the oil content of the whole berry was determined.

Another 30g of whole berries were oven dried to dryness at 130 degrees Celsius for approximately 3 hrs. The seeds were manually separated from the pulp. The oil content in the seed and the pulp were determined on a mechanical extractor system by using hexane as an extraction solvent.

Oil distribution in the major berry components was determined.

1.3. Non-solvent oil isolation trials

Approximately 1.4kg of berries were processed according to the processing flow chart presented in Figure 1. Creekside Acres outlined the process. Basically, it involved steps to spray wash frozen whole berries, press the berries on a lab scale cold press (Type NMRV 90, Motovariom S.P.A., Italy) to obtain press-cake (seeds and pulp) and crude juice. The crude juice was then centrifuged twice at 5000 * g for 15 min to obtain oil-enriched fractions. The press-cake was washed with water, followed by centrifugation to extract more oil. The oil-enriched fractions were analyzed for oil content and fatty acid composition. Oil yield based on the current non-solvent isolation method was determined.

2. Pilot scale solvent oil extraction trials

In order to increase extraction efficiency, berries were dried and milled before they were solvent (hexane) extracted for oil.

Approximately 96.0 kg of berries were processed. The berries were filled in perforated trays to 1-3 cm in height, and convection tunnel dried at 55-70 degrees Celsius to less than 5% moisture. The dried berries were then Fitz milled (Model DAS06, Fitzpatrick Co., Elmhurst, Illinois) at a feed rate of 30-40 kg/hr to break down the seed and the pulp. The Fitz mill was equipped with a 0.125" screen. The milled berries were solvent (hexane) extracted on a Soxlet Extractor for 4 hrs. Due to limited capacity of the extractor, a total of three extraction batches were carried out. Each batch used 4.0-4.5 kg of milled berries, and 11-12 kg of fresh hexane. At the end of the extraction, solvent-oil was vacuum desolventized at 60 degrees Celsius in order to reduce as much as possible the level of hexane in the oil. Crude sea buckthorn oil obtained was analyzed for specific gravity, viscosity, pH, fatty acid composition, carotenes, tocopherols and vitamin E content, and hexane residual level. Oil yield obtained from the pilot scale trials was calculated.

RESULTS AND DISCUSSION

Basic information on the sea buckthorn berry and the results obtained from the solvent and the non-solvent oil isolation trials are discussed as follows:

1. Composition of sea buckthorn berry

The lab studies found that the original frozen sea buckthorn berry contained approximately 4.4%stems. In weight basis, the berry contained 83,6% moisture or juice, 12.05% pulp, and 4.26% seeds.

The moisture content of 83.68% appeared to be in the high range compared to those reported (73.6-85.3%) in the literature (Li and Wang, 1998). Growing location and conditions, maturity, season of harvesting, and variety itself would influence the moisture content of sea buckthorn berry. Theoretically, it would be more efficient to process berries with low moisture contents for oil extraction, as they would contain more oil per berry weight basis. However, the relationship between oil quality and berry moisture is unknown.

2. Oil contents and fatty acid composition

Analytical results showed that the whole berry contained 2.37% oil on wet basis, or 14.53% oil on dry basis. The seeds and pulp contained, on dry basis, 11.62% and 15.11% oil respectively.

Based on mass balance calculations, between 75-80% of the total oils found in the pulp, and between 20-25% of the total oils are located in the seeds. Clear juice contained only a trace amount of oil.

Fatty acid composition in the seed and pulp oils revealed that the seed oil is highly unsaturated, with a total of 60.80% of fatty acids making up the oil being linoleic (C18:2) and linolenic acids (C18:3) (Table 1). Pulp oil is more saturated with 33.08% of the fatty acids making up this oil being palmitic acid (C16), and 35.29% are palmitoleic acid (C16:1).

The fatty acid composition determined on the current sea buckthorn sample corresponded largely to those published in the literature. Lu (1992) reported that, depending on the variety of sea buckthorn berries, up to 73% of total fatty acids. Beveridge *et al.* (1999) claimed that pulp oil contained about 38% of palmitic acid, and 14-50% of palmitoleic acid.

The difference between seed and pulp oils seems to lie in the relatively high content of C16 fatty acids in the pulp and relatively high proportion of C18 fatty acids in the seed oil.

The next most important fatty acid is oleic acid (C18:1) at 16.33% for seed oil, and 10.54% for pulp oil, underscoring the highly unsaturated nature of the oils in both the pulp and seed. This is important from a consideration of potential shelf life because oxidation can be expected to be a major mechanism of degradation.

TABLE 1. FATTY ACID COMPOSITION OF SEED AND PULP OILS

Fatty Acids	Seed Oil (%)	Pulp Oil (%)
C12		0.07
C14	0.24	0.70
C14:1		0.08
C15	0.10	0.08
C16	11.2	33.08
C16:1	7.30	35.29
C16:2	0.53	2.58
C18	2.53	1.22
C18:1	16.33	10.54
C18:2	30.04	13.92
C18:3	30.76	1.55
C20	0.41	0.25
C20:1	0.25	
C20:2	0.04	
C22	0.11	0.10
C24	0.05	0.13

3. Yield and properties of the oil obtained from non-solvent isolation trials

Following the cold pressing, the yield of crude juice was calculated at 51%, and press cake yield was at 47%. The juice yield was lower than those (61.5-79.4%) obtained from various pilot scale pressing trials (Li and Wang, 1998). The lab press used is mainly designed to process oil seeds, which are relatively low in moisture contents. Its efficiency and effectiveness would be reduced when processing "watery" materials, such as sea buckthorn berries. Materials with high moisture contents would not be able to generate sufficient backpressure in the press, which are required to effectively extract the liquid from the solid meals. A hydraulic press would be more suitable in order to obtain high juice yields from the products.

As a result of centrifugation of the crude juice, a total of four layers were visibly distinguished. The top layer contained 40-66% of oil; the 2nd emulsion layer contained 20-25% of oil. The 3rd layer was clear juice, and contained trace amounts of oil. The bottom 4th layer was pulp and seeds.

Following the water wash of the emulsion and pulp and seeds, more oil was extracted (Figure 1).

Based on 1401.7g of berries processed, combined oil-enriched fractions (oil #1 and oil #2, Figure 1) obtained weighed 5.3 g, and contained approximately 50%, or 2.65 g of oil. The oil yield using the current non-solvent isolation method was calculated at 0.19% (weight of oil/weight of starting berries, x 100%).

The low oil yield would be attributed to low oil content in the whole fruit and poor solvent (water) system used. Since most of the oils in sea buckthorn berry are the neutral lipids, therefore a neutral or low polarity solvent such as hexane would be more efficient for oil extraction. The two emulsions obtained following centrifugation of the juice weighed a total of 62.4 g(Figure 1), and contained approximately 12.5 g of oil. However, further purification of the oil by breaking up the emulsion would require extremely high force centrifuges. Based on mass balance calculations, between 14-16 g of oil remained in the final solids fractions, which contained pulp, seeds and moisture.

As most of the seeds remained sound and intact, the oil obtained from the lab trials were therefore essentially the pulp oil, as illustrated by its fatty acid composition (Table 2).

It was concluded that the non-solvent isolation method was not efficient in obtaining high oil yields. Isolation of the oil from the seeds would remain a problem if organic solvent(s) are not used. The solvent method would be an alternate choice to extract the oil from sea buckthorn berry.

4. Yield and properties of the oil produced from solvent extraction trials

Pilot scale oil extraction trials involved drying of whole fruit to less than 5% moisture, followed by milling, oil solvent (hexane) extraction and desolventization to obtain final oil product.

4.1 Drying and milling

Drying of the berries from initial 83-85% to less than 5% moisture required 13-15h. Two drying batches were conducted, and 14.02 kg of dried berries were obtained based on 96.0 kg of berries processed.

The berries were dried with almost no loss of oil-enriched berry components, which included seeds, pulp and fruit particles. Only an insignificant amount of juice was lost through the perforated drying trays.

Due to the heat treatment, the drying process resulted in a brown product as compared to its original yellowish color. Never the less, the Tunnel dryer was among the best choices of dryers for berry drying. The use of other dryers would either result in high losses of materials (such as fluid bed dryer), or high operation costs (such as freeze-dryer).

Following the Fitz milling, dried pulp and seeds were reduced in size which would facilitate solvent penetration during the oil extraction process.

4.2 Solvent extraction

Following 4 h of hexane extraction, the mixture of extracted oil and hexane was desolventized at 60oC under the vacuum. The level of hexane in the final oil was reduced to 692 PPM.

Crude oil obtained weighed 2.0 kg, which represented an oil yield of 2.08% based on 96.0 kg of berries processed.

The oil is orange- reddish in color, which reflects high levels of carotenoids (Table 2), as well as a number of pigmented compounds removed during solvent extraction. The color of carotenoids is the result of the presence of a conjugated double bond system in the molecules.

The crude oil had a specific gravity of 0.90 g/ml, pH 3.15, and viscosity of 62.5 cps at 25oC (Table 2).

Fatty acid composition of the crude oil is profiled (Table 2) between that of the seed and pulp oils (Table 1). It is actually closer to that of the pulp oil, as the pulp oil makes up 75-80% of the total oils in the sea buckthorn berry.

TABLE 2. PROPERTIES OF THE OILS OBTAINED FROM LAB AND PILOT SCALE TRIALS

	Oil obtained from lab scale non-solvent extraction trials	Crude oil obtained from pilot scale solvent extraction trials
Fatty Acids (%)		
C12	0.07	0.06
C14	0.67	0.59
C16	32.25	28.51
C16:1	36.31	30.49
C16:2	2.71	1.97
C18	1.10	1.42
C18:1	10.26	11.06
C18:2	14.11	16.59
C18:3	1.68	8.58
C20	0.23	0.28
C20:1	0.09	0.07
C22	0.05	0.10
C24	0.08	0.08
SPECIFIC GRAVITY		0.90g/ml
***Viscosity**		62.5 cps
pH		3.15
Carotenes (ug/g)		
Alpha carotene		41.8
Beta carotene		414.8
Lycopene		252.0
Tocopherols (mg/g)		
Alpha		1.25
Beta		0.10
Gamma		0.17
Delta		<0.05
HEXANE		692 ppm

* viscosity was measured on a Wells-Brookfield Cone/Plate Viscometer at 25 degrees Celsius, cone#cp-42, speed = 6 rpm, shear rate =23 s-1

The crude oil contained 1.57 mg/g of tocopherols, with 79.7% being the alpha-tocopherols, or vitamin E. As natural antioxidants, they prolong the shelf life of many foods containing fat or oil. The tocopherols and vitamin E contents determined on the crude oil sample were lower compared to those reported. Zhang et al. *1989) reported that vitamin E content in seed and pulp oil were at 0.61-1.13 mg/g and 1.62-2.55 mg/g, respectively. Part of the tocopherols and vitamin E would be lost during the drying and solvent extraction stages.

CONCLUSIONS

Non-solvent isolation protocol resulted in low oil yields, largely due to the difficult water extraction and separation of the oil from the pulp and seeds. Technically, the yield and purity would be improved if high force centrifuges were used. However it would remain inefficient to extract the oil from the seeds without using organic solvent(s) or having the seeds ground and treated with special enzymes. The oils obtained would be essentially the pulp oil based on the fatty acid composition evidence.

The solvent extraction method was able to obtain high oil yields. However it required pre-drying and grinding of the berry prior to the extraction process. Heat treatment during the drying and milling stages causes the berry to lose part of its valuable components, such as tocopherols and vitamin E, which are considered as natural anti-oxidants. The heat treatment also darkened the color of the finished oil product, partially due to the formation of colored compounds.

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